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A CUTTING BLADE FOR A SURGICAL INSTRUMENT**BACKGROUND TO THE INVENTION**

THIS invention relates to a cutting blade for a surgical instrument in which the cutting blade is formed of a hard transparent, crystalline material, such as diamond sapphire or garnet, on the surface of which is provided a layer of fluorine atoms chemically bonded to the surface.

Surgical blades are extremely sharp in order to minimise tissue damage along a line of incision. In order to achieve the desired sharpness of a cutting blade materials of choice for the manufacture of cutting blades are hard materials of a crystalline nature, such as diamond or sapphire.

During use blood and other bodily fluids and materials often stick to the facets of a cutting blade thereby reducing its effectiveness. It is known to prevent this from happening or at least reduce the sticking effect and facilitate cleaning of the blade by, for instance, wiping the blade with a suitable material or sticking it into a block of suitable plastic foam, for example polystyrene.

The problem of blood sticking to or coagulating on the surface of a cutting blade may be aggravated under conditions where coagulation of blood is promoted. This may be caused by deliberate heating of the surgical blade to induce coagulation; by high intensity light sources used in conjunction with the blade or by the simultaneous use of a laserbeam, either through the cutting blade or applied separately.

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South African provisional patent application no. 99/4256, also filed by the applicant in this instance, describes a cutting blade for a surgical instrument in which the cutting blade is formed of diamond and laser radiation is transmitted through the blade in order to provide a cauterisation effect along a line of incision. This earlier application is incorporated herein by reference. The laser radiation passing through the cutting blade which forms the subject of this invention would cause heating of the blade which encourages blood sticking and coagulating on the surface of the blade.

SUMMARY OF THE INVENTION

According to the invention there is provided a method of forming a protective layer of fluorine atoms on a cutting blade of a surgical instrument in which the blade is formed of hard, transparent, crystalline material, such as diamond, sapphire or garnet, the method comprising the steps of:

- a) placing the blade in a plasma reactor;
- b) plasma cleaning the blade; and
- c) coating the blade in a plasma of carbon fluoride (C_nF_m) gas.

Preferably, the carbon fluoride (C_nF_m) containing gas is C_3F_8 , alternatively C_2F_4 or C_2F_6 .

The method may include the step of chemically cleaning the blade.

Typically, the coating takes place at a pressure of 0.01 to 2 mbar, for a period of 30 to 180 minutes and at a power level of 50 to 2000 watts.

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Conveniently, the cleaning takes place in a plasma of air, oxygen, argon or a mixture thereof.

According to a second aspect of the invention there is provided a cutting blade for a surgical instrument, the cutting blade being formed of a hard, transparent, crystalline material, such as diamond, sapphire or garnet, on the surface of which is provided a protective layer of fluorine atoms formed in accordance with the method described above.

Preferably, the blade is formed of natural, monocrystalline synthetic or polycrystalline synthetic diamond or sapphire.

According to a third aspect of the invention there is provided a method of forming a protective layer of fluorine atoms on a blade of a surgical instrument characterised in that the method comprises the step of immersing the blade into a solution of a fluoroaliphatic silyl ether.

The method is typically performed on a blade formed of diamond.

Preferably, the method includes the step of curing the layer at a temperature in excess of 200° C.

The method may include a step of forming a hydroxyl terminated surface on the blade before immersion of the blade into a solution of a fluoroaliphatic silyl ether.

The method may also include the step of forming an intermediate silicon or Ti layer on the surface of the blade prior to immersion of the blade into a solution of a fluoroaliphatic silyl ether. The Si layer preferably has a thickness less than 50 nm.

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Various embodiments of the invention are described in detail in the following passages of the specification. The described embodiments are merely illustrative of how the invention might be put into effect and should not be seen as limiting on the scope of the invention.

DESCRIPTION OF AN EMBODIMENT

In general terms this invention relates to a method of forming a protective layer of fluorine atoms on a cutting blade for a surgical instrument in which the surgical blade is formed of a hard, transparent, crystalline material such as diamond, sapphire or garnet. The purpose of the layer is to reduce the sticking effect of blood and bodily fluids and materials to the blade during use. The layer should be of minimum thickness to minimise the reduction in sharpness of the blade. It is envisaged that this may be achieved according to the invention either by minimising the thickness of the layer (in the extreme case one atomic layer of fluorine) or by polishing a micro facet on one or both sides of the cutting edge after the coating has been applied.

The method of the invention is in essence a plasma coating method involving the following steps:

1. Chemically cleaning the blade.
2. Placing the cutting blade in a plasma reactor.
3. Plasma cleaning of the blade. This is done in a plasma of air, oxygen, argon or a mixture thereof for 5 to 20 minutes at approximately 1 mbar pressure and a power level of approximately 500 watts. The power is switched on at a duty cycle of 5 % to 50 % to prevent overheating. This cleaning step is essential if good adhesion of the fluorine containing layer

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is to be achieved.

4. Coating the blade in a plasma of C_3F_8 . The process conditions of this coating step are a pressure of 0.01 to 2 mbar for a period of 30 to 180 minutes at a power level between 50 and 2000 watts.

The above description is a description of one method of putting the process of the invention into effect and of variations on the specific process conditions described above.

Two different approaches may be used in the process described above:

1. The chemical structure of the diamond or other hard, crystalline material is modified such that it terminates with fluorine atoms, instead of the more usual hydrogen and/or oxygen. This can be achieved by exposing the surface of the material, such as diamond, to atomic fluorine at a range of temperatures, between 273 and 573K. The preferred deposition method for the fluorine atomic layer onto the surgical blade is plasma treatment. In this method the surgical blade is exposed to a plasma excited in an atomic fluor generating substance such as SF_6 , NF_3 , HF or F_2 . Argon may be introduced into the plasma to reduce the deposition rate to controllable levels.
2. The surface is coated with a fluorocarbon polymer layer. This can be achieved by the known technique of plasma polymerization using precursors such as tetrafluoroethene. This process is described in the article entitled "Fundamentals of Plasma Chemistry and Technology" H.V. Boenig, Pub Technomatic, 1988 and the other references referred to in this document, which are all

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incorporated herein by reference.

The preferred deposition method for the fluorocarbon polymer layer onto the surgical blade is plasma treatment. In this method the surgical blade is exposed to a plasma excited in a carbon fluoride gas. Argon may be introduced into the plasma to reduce the deposition rate to controllable levels.

The thickness of the fluorocarbon polymer layer created by this process is a function of the time for which the blade is subjected to the process. The coating thickness can vary from a few nanometers to hundreds of nanometers. Thinner coatings are more desirable so as not to blunt the cutting edge of the blade and limit laser light absorption.

The polymer is deposited from a plasma excited from one of the following gases:

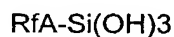
C_2F_4 , C_2F_6 , C_3F_8 .

The layer thickness is typically between 5 nanometers and 10 microns. A micro facet of between 5 and 50 microns is polished on one or both sides of the cutting edge after the layer has been formed.

In addition to the methods described above other processes may also be used to achieve the desired layer of fluorine atoms on the surface. One such method is to heat the blade in a C_2F_4 environment. This induces polymerisation of the C_2F_4 on the hot surfaces to form a layer of fluorine atoms.

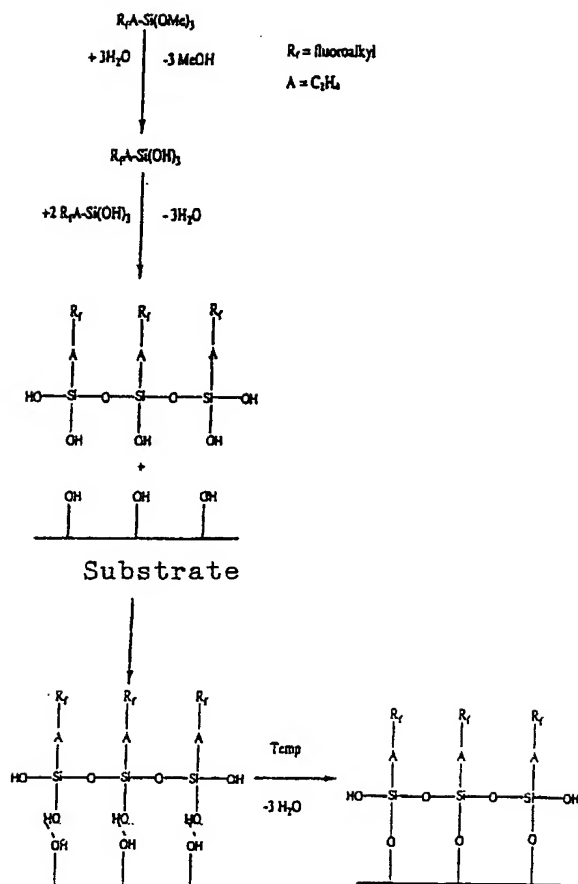
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The layer of fluorine atoms on the surface may also be applied in other ways. For example, the fluorine atoms may be chemically bonded to the diamond surface by attaching a chemically reactive group to a fluorinated alkane group. Such a fluorinated alkane is a molecule in which fluorine atoms replace hydrogen atoms in a (usually linear) carbon chain. This is an inert molecule and a polymerised variant is the basis for the product known by the proprietary name of "Teflon". By attaching a chemically reactive group to the fluorinated alkane it can be bonded to the diamond surface. An example of such a chemically reactive group is a group containing SiOH, which can bond to a surface, which is hydroxyl (-OH) terminated. The SiOH group can bond to the hydroxyl terminated surface by splitting off a water molecule, thus forming a fluorinated_tail-Si-O-Si-surface bond. An example of this type of coating material is fluoroaliphatic silyl ethers, whose generic chemical formula is given below.



A schematic representation of this reaction is provided overpage.

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where R_f is a fluorinated alkyl group, A is C_2H_4 , and $Si(OH)_3$ is the active bonding group. In this case one of the OH groups can bond to the surface, while the others bond to other fluoroaliphatic silyl ether molecules, thus forming a network.

An example of a fluoroaliphatic silyl ether is the product sold under the brand name FC405/60 the 3M company. Here the fluoroaliphatic silyl ether molecules are dissolved in a solvent such as an alcohol (e.g. isopropanol).

By further diluting the solution with isopropanol so that a concentration of the fluoroaliphatic silyl ether molecules is obtained of less than 1% (e.g. adding 0.5 ml of coating fluid to 60 ml of isopropanol) and adding acetic acid to give a value of the pH of between 4 and 5.5, a layer of fluorine atoms can be applied to the surface of a diamond blade by dipping it in the solution for approximately 3 minutes. It is recommended that the solution

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be stirred ultrasonically to establish good contact of fresh coating fluid with the surface of the blade. The blade is drawn out of the coating fluid and the remaining layer of coating solution is rinsed off with isopropanol. The coating is then allowed to cure at an elevated temperature. Although the product information supplied by the manufacturer of the fluoroaliphatic silyl ether fluid states that curing should take place for 5 minutes at 110° C, it has been found that a coating with better scratch and rubbing resistance and better adherence to the diamond blade surface can be achieved by using a temperature of 235° C for approx. 1 hour.

In respect of diamond there is an additional difficulty in chemically bonding the coating material to its surface. This is due to the fact that in general a diamond surface does not have hydroxyl groups attached to its surface. Methods of applying a hydroxyl-coated surface are therefore part of this invention. One such method achieves this by immersing the diamond blade in a bath of molten alkali hydroxide, such as sodium hydroxide or potassium hydroxide or mixtures of these with sodium- or potassiumnitrate for periods of up to one hour. Another, though less effective, method is the application of a microwave discharge in water vapour to the diamond blade surface. This dissociates water molecules and forms OH radical groups in vapour form, which can attach to the diamond surface. The discharge, however, will also generate other radical species which can attach to the surface as well, and thus occupy some bond sites, which are then not available to hydroxyl groups. This latter method results in a partially hydroxyl covered surface. Other methods include application of an interfacial layer, such as titanium (Ti), chromium (Cr). The layer can be hydroxyl terminated by immersion in dilute NaOH. It is also possible to attach the fluoroaliphatic silyl ether to the metal surface directly by dipping the freshly coated surface into the coating liquid.

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Formation of a hydroxyl-terminated Si layer can also be achieved by immersing the diamond blade in a dilute (approx. 10%) solution of NaOH in water for approx. 3 minutes at approx. 90-100° C, followed by rinsing in deionized water, dipping in a concentrated (>20%) solution of HCl in water, rinsing again in deionized water, rinsing in ethanol and finally isopropanol and then allowing the blade to dry. After this step the blade is immersed in the coating liquid and the coating is applied as described above.

The preferred manner of attaching coating molecules to a diamond surface has been to coat the surface of the diamond with a thin layer of silicon (Si). This layer, which is typically less than 50 nm thick forms a chemical bond with the diamond by the formation of SiC. A larger thickness of the Si layer is disadvantageous as it will result in a reduced transmission of the infrared radiation out of the blade and concomitant absorption of the radiation in the blade, leading to a reduced cauterising effect in the tissue and/or heating of the blade and extra sticking of tissue or blood to the blade. For applications where light is not required to exit the Si layer the layer may be applied thicker or another interfacial layer may be applied.

The cutting blades to which this process may be applied are formed of hard, transparent crystalline material. Typically this material is natural, monocrystalline synthetic or polycrystalline synthetic diamond or sapphire.

However, other materials could also be used such as hard crystalline simple oxides such as zirconia (ZrO_2), yttria (Y_2O_3), garnets, most notably YttriumAluminumGarnet, LutetiumAluminumGarnet, vanadates and aluminumoxides (such as YttriumAluminumOxide.) Other hard infrared transparent crystals which may also be appropriate for the process are, orthosilicates.

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The method which forms the subject of this invention can be applied to a wide range of cutting blades operating in a range of laser wavelengths, such as those which are described in South African provisional patent application no.99/4256.

CLAIMS:

1. A method of forming a protective layer of fluorine atoms on a cutting blade of a surgical instrument in which the blade is formed of hard, transparent, crystalline material, the method comprising the steps of:
 - a) placing the blade in a plasma reactor;
 - b) plasma cleaning the blade; and
 - c) coating the blade in a plasma of carbon fluoride (C_nF_m) gas.
2. A method according to claim 1, wherein the blade is formed of diamond, sapphire or garnet.
3. A method according to either claim 1 or claim 2, wherein the carbon fluoride (C_nF_m) gas is C_3F_8 , C_2F_4 or C_2F_6 .
4. A method according to any one of the preceding claims, wherein the method includes the step of chemically cleaning the blade.
5. A method according to any one of the preceding claims wherein, the coating takes place at a pressure of 0.01 to 2 mbar, for a period of 30 to 180 minutes and at a power level of 50 to 2000 watts.
6. A method according to any one of the preceding claims, wherein the cleaning takes place in a plasma of air, oxygen, argon or a mixture thereof.

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7. A cutting blade for a surgical instrument, the cutting blade being formed of a hard, transparent, crystalline material, on the surface of which is provided a protective layer of fluorine atoms formed in accordance with the method described above.
8. A cutting blade according to claim 7, wherein the cutting blade is formed of diamond, sapphire or garnet.
9. A cutting blade according to claim 7, wherein the blade is formed of natural, monocrystalline synthetic or polycrystalline synthetic diamond or sapphire.
10. A method of forming a protective layer of fluorine atoms on a blade of a surgical instrument characterised in that the method comprises the step of immersing the blade into a solution of a fluoroaliphatic silyl ether.
11. A method according to claim 10, wherein the blade is formed of diamond.
12. A method according to either claim 10 or claim 11, wherein the method includes the step of curing the layer at a temperature in excess of 200° C.
13. A method according to any one of claims 10 to 12, wherein the method includes a step of forming a hydroxyl terminated surface on the blade before immersion of the blade into a solution of a fluoroaliphatic silyl ether.

14. A method according to any one of the preceding claims, wherein the method includes the step of forming an intermediate silicon layer on the surface of the blade prior to immersion of the blade into a solution of a fluoroaliphatic silyl ether.
15. A method according to claim 14, wherein the Si layer has a thickness less than 50 nm.

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(54) Title: A CUTTING BLADE FOR A SURGICAL INSTRUMENT

(57) Abstract: This invention relates to a method of forming a protective layer of fluorine atoms on a cutting blade of a surgical instrument in which the blade is formed of a hard, transparent, crystalline material such as diamond, sapphire or garnet. According to the method the blade is placed in a plasma reactor, the blade is then plasma cleaned and coated with a plasma of carbon fluoride gas. The invention also relates to a method of forming a protective layer of fluorine atoms on a blade for surgical instruments in which the blade is immersed into a solution of fluoroaliphatic silyl ether.

PATENT APPLICATION
Docket No. 42377-00012

**RULES 63 AND 67 (37 C.F.R. 1.63 and 1.67)
DECLARATION AND POWER OF ATTORNEY**

FOR UTILITY/DESIGN/CIP/PCT NATIONAL APPLICATIONS

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;
and

I believe that I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled: A Cutting Blade for a Surgical Instrument, the specification of which: (mark only one)

- ☐ (a) is attached hereto.
- ☒ (b) was filed on January 23, 2002 as Application Serial No. 10,048,131 and was amended on _____ (if applicable)
- ☐ (c) was filed as PCT International Application No. PCT/_____ on _____ and was amended on _____ (if applicable).
- ☐ (d) was filed on _____ as Application Serial No. _____ and was issued a Notice of Allowance on _____.
- ☐ (e) was filed on _____ and bearing attorney docket number _____.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims as amended by any amendment referred to above or as allowed as indicated above.

I acknowledge the duty to disclose all information known to me to be material to the patentability of this application as defined in 37 CFR § 1.56. If this is a continuation-in-part (CIP) application, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose to the Office all information known to me to be material to patentability of the application as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this CIP application.

I hereby claim foreign priority benefits under 35 U.S.C. § 119/365 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate filed by me or my assignee

[illegible]

<u>Number</u>	<u>Country</u>	<u>Month/Day/Year Filed</u>	<u>Date first laid-open or Published</u>	<u>Date patented or Granted</u>	<u>Priority Claimed</u>	<u>Yes</u>	<u>No</u>
99/4910	South Africa	30 July 1999			X		


PRIOR U.S. OR PCT APPLICATIONS

I hereby appoint the attorneys listed on Attachment A, all of the firm of **JENKENS & GILCHRIST, P.C.**, 3200 Fountain Place, 1445 Ross Avenue, Dallas, Texas 75202-2799, as my attorneys and/or agents, with full power of substitution and revocation, to prosecute this application, provisionals thereof, continuations, continuations-in-part, divisionals, appeals, reissues, substitutions, and extensions thereof and to transact all business in the United States Patent and Trademark Office connected therewith, to appoint any individuals under an associate power of attorney and to file and prosecute any international patent application filed thereon before any international authorities, and I hereby authorize them to act and rely on instructions from and communicate directly with the person/assignee/attorney/firm/organization who/which first sent this case to them and by whom/which I hereby declare that I have consented after full disclosure to be represented unless/until I instruct them in writing to the contrary.

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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